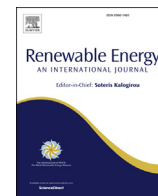




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## Review

## Extrusion as a pretreatment for lignocellulosic biomass: Fundamentals and applications

Aleta Duque<sup>\*</sup>, Paloma Manzanares, Mercedes Ballesteros

Biofuels Unit, Energy Department-CIEMAT, Avda. Complutense, 40, 28040, Madrid, Spain

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## ABSTRACT

Pretreatment of lignocellulosic biomass is an essential step to obtain sugars from such biomasses, aimed at breaking the recalcitrant structure of lignocellulose and facilitate the access of enzymatic hydrolytic agents to carbohydrates. Among the variety of pretreatment technologies that have been investigated in the past years, extrusion is a promising thermo-mechanical pretreatment. It is a continuous process, highly versatile, having good mixing and heat transfer capabilities and being able to operate at high solids loadings. However, its energetic and economic feasibility needs still to be evaluated before it can be an actual alternative to conventional biomass pretreatments.

With the aim to provide a further insight into extrusion pretreatment, this work reviews its fundamentals and the influence of the most relevant operation variables (screw profile and speed, residence time, temperature, liquid to solid ratio and downstream operations) on extrusion performance, analyzed according to the existing bibliography. To complete this overview, the effects of extrusion on lignocellulosic biomass structure, composition and enzymatic digestibility are studied, with a focus on the performance of catalyzed extrusion.

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Abbreviations: EH, enzymatic hydrolysis; L/D, length to diameter ratio; L/S, liquid to solid ratio inside the extruder; LCB, lignocellulosic biorefineries; LB, lignocellulosic biomass; SEM, Scanning electron microscopy; SSA, Specific surface area.

<sup>\*</sup> Corresponding author.

E-mail address: [aleta.duque@ciemat.es](mailto:aleta.duque@ciemat.es) (A. Duque).

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## 1. Introduction

Lignocellulosic biorefineries (LCB) success will be based on maximizing the value of complex biomass materials by extracting and valorizing all components while minimizing the processing needs [1]. LCB relies on an efficient separation or fractionation process (pretreatment) that results in the separation of main components (cellulose, hemicellulose and lignin), to be processed into biofuels and biochemicals. Carbohydrate fraction is mostly used for the production of fermentable carbohydrates by enzymatic conversion, resulting in a stream of fermentable sugars that can be sent directly to the production of biofuels, bioproducts and biochemicals. Lignin represents a valuable source of aromatic chemicals or can be used as a fuel in the lignocellulosic biorefinery.

Extensive literature on pretreatment technologies to fractionate lignocellulosic biomass (LB) into its main constituents has been published in the last years [2–4]. Commercial methods are based on the use of high pressure and temperature, with or without chemical agents. Extrusion, however, stands out as a technology that can effectively fractionate lignocellulosic biomass at mild temperature and lower chemicals, preventing the formation of inhibitory by-products occurring at more severe conditions [5]. Extrusion is a relatively novel pretreatment, in which the material is submitted to mixing, heating and shearing, resulting in physical and chemical modifications [6]. It is a continuous process characterized for its ability to provide high shear, rapid heat transfer, and efficient and fast mixing [7]. Furthermore, extrusion is able to work at high solid concentrations and it is a highly versatile pretreatment that can be used alone, or combined with both chemicals (reactive extrusion) and other pretreatment methods. It can be run at moderate temperature and pH conditions, which reduce sugars degradation and avoids the generation of toxics [5].

As a consequence of these favorable features, an increasing number of research studies have been published, dealing with the effectiveness of extrusion to produce fermentable sugars from a wide range of lignocellulosic biomasses. The results vary depending on the particular conditions of the pretreatment and the biomass used, but in all cases there is an improvement of the sugar production yield from extruded materials (hereinafter, extrudates) compared to the raw material.

A look at the existing bibliography about LB extrusion shows that agricultural wastes and herbaceous biomasses such as corn stover [8–11], are feedstocks mostly used for this pretreatment. These kind of herbaceous materials is usually preferred because they are softer and easier to extrude; however, extrusion of woody biomasses such as poplar [12], hardwood [13], Douglas fir and eucalyptus [14,15], or pine wood chips [16], have also resulted, under proper conditions, in substrates with enhanced enzymatic digestibility.

Although some research has been carried out about extrusion as sole pretreatment for lignocellulosic biomass [8–11,17–20], there is a clear trend towards using extrusion in combination with other kinds of pretreatments. In fact, the efficiency of extrusion pretreatment and, therefore, its effect on the enzymatic digestibility, seems to be limited when no chemical agents are used [21]. This topic will be developed later on this paper under paragraph 4.

On the other hand, the important prospective shown by

extrusion as LB pretreatment, has led to a number of processes having been patented for the treatment of lignocellulosic substrates, as can be seen in Table 1. First patents were issued in the late 80' with the use of alkaline extrusion [22], or extrusion in combination with other pretreatments such as steam-explosion [23] or acid hydrolysis [24]. From that time on, other process configurations involving extrusion have been patented, more intensively in the last 10 years [25–32].

All patents shown in Table 1 claim advantages of the disclosed processes (all of them comprehending extrusion) compared to the reference pretreatments; reduced costs [24–26], time [22,27,28] and heat consumption [22,29], and production of substrates with increased hydrolysis efficiency [25,27,30,31] and less degradation of hemicelluloses [24,33].

Coinciding with the increasing attention drawn by extrusion pretreatment, some reviews have recently appeared [33–35] and the present work aims at contributing to a more comprehensive study of extrusion pretreatment by covering all aspects of the technology. Thus, this paper aims at providing an insight on the fundamentals and operation of extrusion, analyzing the particular effect of each operation variable on the extrusion performance. In addition, the effects of this pretreatment on the biomass are analyzed based on results from abundant research effort carried out during the last years on numerous lignocellulosic substrates at different extrusion conditions.

## 2. Extrusion process

Extrusion is a technology that was first used for metal conformation, but soon expanded to many other applications in several fields such as ceramics, rubber, food processing, chemical, polymer, composites and energy industries. Extrusion is a thermo-mechanical process that is based on the action of one or two screws that spin into a tight barrel, which is equipped with temperature control (Fig. 1). This action produces high shearing forces between the raw material, the screw and the barrel that lead to locally temperature and pressure rise along the extruder.

The main distinction among extrusion machines is made between single-screw and twin-screw extruders. Single-screw extruders are made of one single solid piece, while twin-screw are comprised of small pieces called screw elements that are assembled in the shafts. In twin-screw extruders there are three main types of screw elements that can be used: kneading blocks, reverse screws and conveying screws (Fig. 2). Additionally, twin-screw extruders can be classified as counter-rotating and co-rotating. The first development of a single screw extrusion machine dates back to the second half of the 19th century. Co-rotating twin-screw extruders started to be used in the 1940's and 50's [36], leading to the licensing of the technology in 1957 [37]. From this point, extruder design kept on evolving, incorporating new features such as the modular technology or the modelling of processes, that allowed to broaden the functionalities of the equipment to, for instance, evaporation, or reaction (from 1970's) [36].

As it can be seen in Figs. 2 and 3, these screw elements have different forms, which produce different effects on the biomass; transport, mixing, shearing and combinations of the three that are responsible for the disintegration of the LB, the good

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